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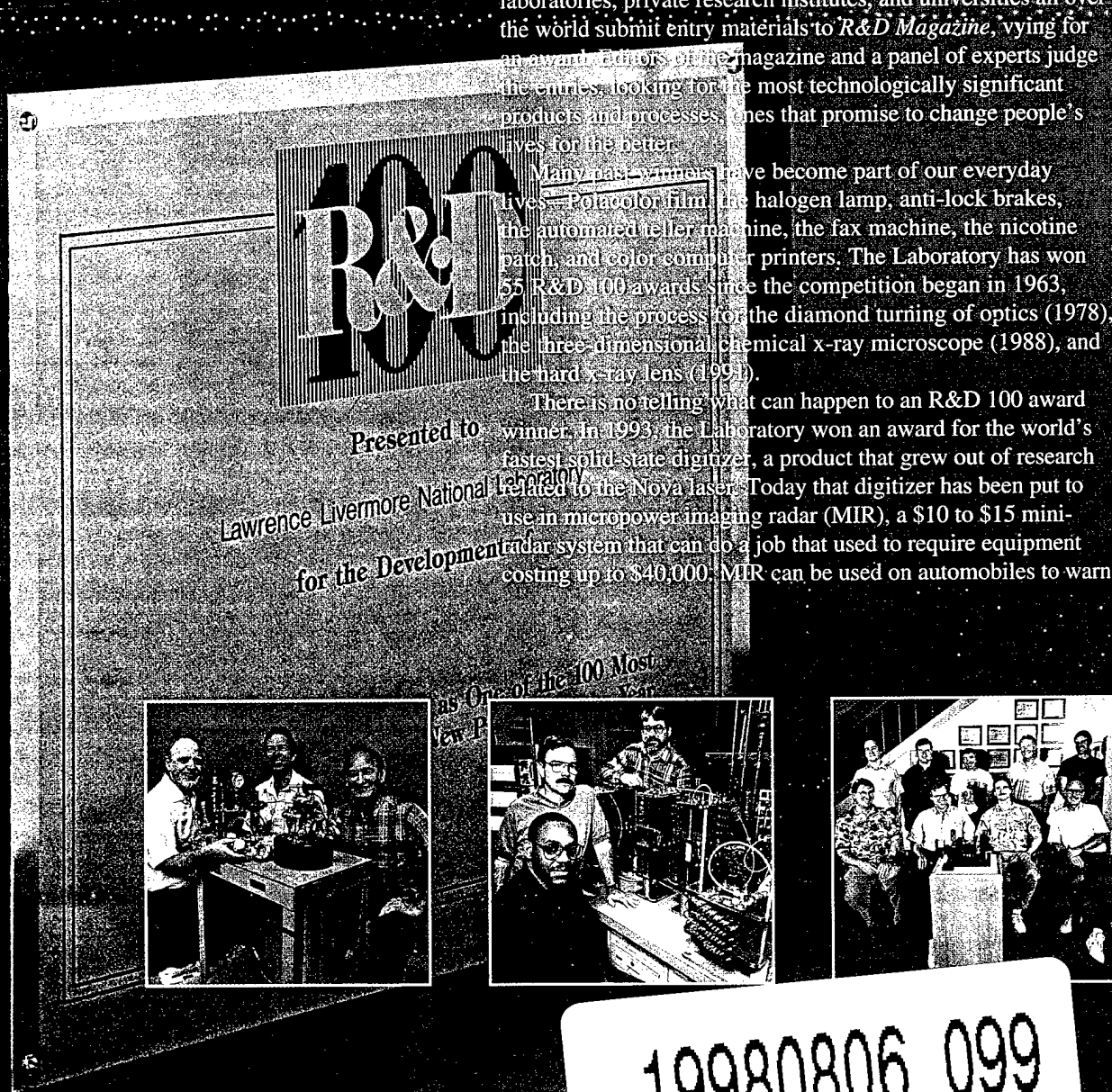
R&D 100 Awards Recognize Five Laboratory Inventions

THE Laboratory has done it again. In receiving five R&D 100 awards, the "Oscars" of applied research, the Laboratory has demonstrated its stature among the premier research institutions in the world.

Every year, scientists from corporations, government laboratories, private research institutes, and universities all over the world submit entry materials to *R&D Magazine*, vying for an award. Editors of the magazine and a panel of experts judge the entries, looking for the most technologically significant products and processes, ones that promise to change people's lives for the better.

Many past winners have become part of our everyday lives—Polaroid film, the halogen lamp, anti-lock brakes, the automated teller machine, the fax machine, the nicotine patch, and color computer printers. The Laboratory has won 55 R&D 100 awards since the competition began in 1963, including the process for the diamond turning of optics (1978), the three-dimensional chemical x-ray microscope (1988), and the hard x-ray lens (1991).

There is no telling what can happen to an R&D 100 award winner. In 1993, the Laboratory won an award for the world's fastest solid-state digitizer, a product that grew out of research related to the Nova laser. Today that digitizer has been put to use in micropower imaging radar (MIR), a \$10 to \$15 mini-radar system that can do a job that used to require equipment costing up to \$40,000. MIR can be used on automobiles to warn



of collisions, can detect buried land mines, and can help rescuers searching for people trapped alive in destroyed or collapsed buildings. The range of MIR applications is so broad that the Laboratory is selling licenses to apply MIR technology in 14 different areas.

This year's awards were given to six teams of Laboratory scientists who made major progress in the fields of aerogels, lasers, mass spectrometry, and electron beam processing.

One is a shared award for two aerogel processes:

- A new injection molding process for aerogels was developed that is similar to the molding process used to manufacture some plastics. This method of mass-producing aerogels is 30 times faster than any other production system, reduces liquid wastes by 40%, and uses 10 times less energy.
- Capacitive deionization with carbon aerogel electrodes provides an economical and efficient method for removing salt and impurities from water. It generates no wastes and is more energy-efficient than competing technologies. It can be used for waste treatment, water purification and softening, and desalination.

Two awards are related to the development of new types of lasers:

- A simple and inexpensive lens device scales up the output power of diode-pumped, solid-state lasers to 250 times that of similar systems. With this increased output energy, these

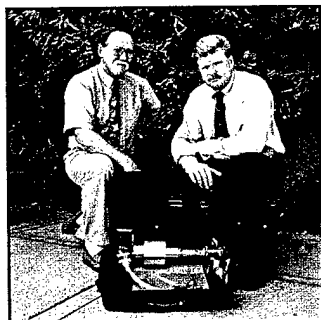
lasers can be used in surgery, to treat infertility, in laser radar, and perhaps eventually in inertial confinement fusion.

- A flashlamp-pumped, solid-state laser with a unique amplifier system is the world's brightest solid-state, average-power laser. Divergence has been reduced almost to its physical limits, and the beam has an extremely narrow bandwidth. The laser is being used to generate x rays for the production of integrated circuits and in long-range laser radar systems.

The final two awards were given for developments in fields as diverse as mass spectrometry and electron beam processing:

- The first hand-portable mass spectrometer, based on the principle of ion-cyclotron motion, combines the ion source and mass analyzer/detector with an integral vacuum system. In spite of its small size and simplicity, the system can achieve sufficient resolution to detect trace compounds or contaminants in air samples.
- A small, inexpensive electron beam gun can be used to process paints, inks, computer floppy disks, medical supplies, and other materials. The beam exits the gun through a new, thin-film window and delivers higher beam energies much more efficiently than large, expensive, conventional electron beam systems.

The group of articles that follows highlights each of these R&D 100 award-winning inventions and introduces you to the men and women of Lawrence Livermore National Laboratory who made them possible.



A Shared Award in Aerogel Process Technology

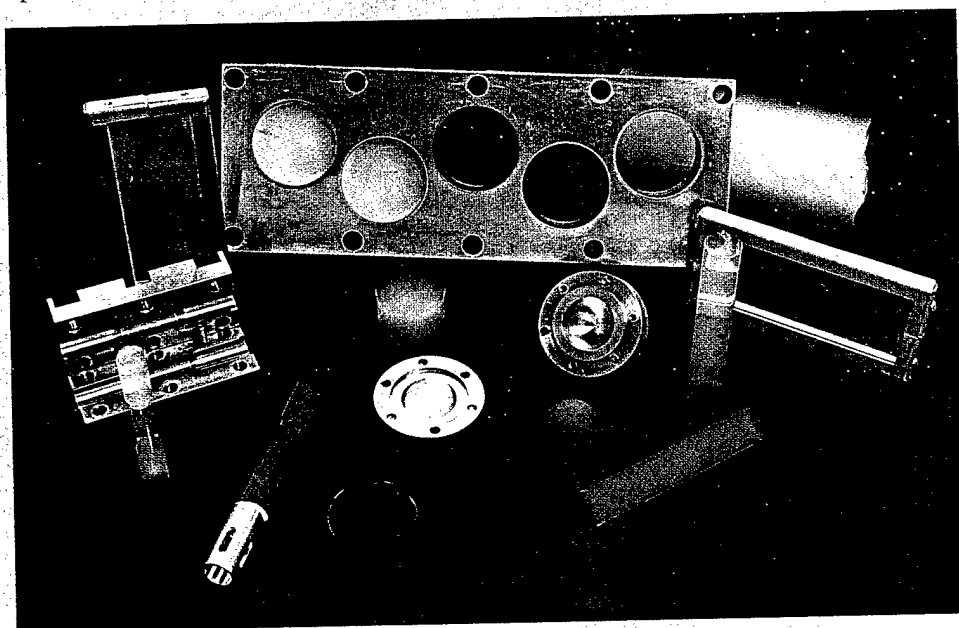
AEROGELS are among the lightest solids known, with some varieties consisting of 99.8% air. Many aerogels are nearly transparent and are called "frozen smoke" for their ghostly appearance. At the same time, of all known solids, aerogels have the highest internal surface areas per gram of material, thanks to their complicated, cross-linked internal molecular structure. An aerogel the size of a grape has the surface area of about two basketball courts. The internal microstructures give aerogels exceptional strength—some aerogels can support 1600 times their own weight.

First made in 1931, silica aerogel, composed mainly of silicon dioxide (sand), is probably the best known type of aerogel. Another type is organic aerogel, made up mainly of carbon and hydrogen atoms. At Lawrence Livermore National Laboratory, one of the world's leading centers of both silica and organic aerogel research and development, scientists attracted international attention early in this decade when they created a silica aerogel some 10 times less dense than the previous lightest version.



Pictured with the injection molding apparatus for rapid production of silica aerogels are, from left to right, Larry Hrubesh, John Poco, and Paul Coronado.

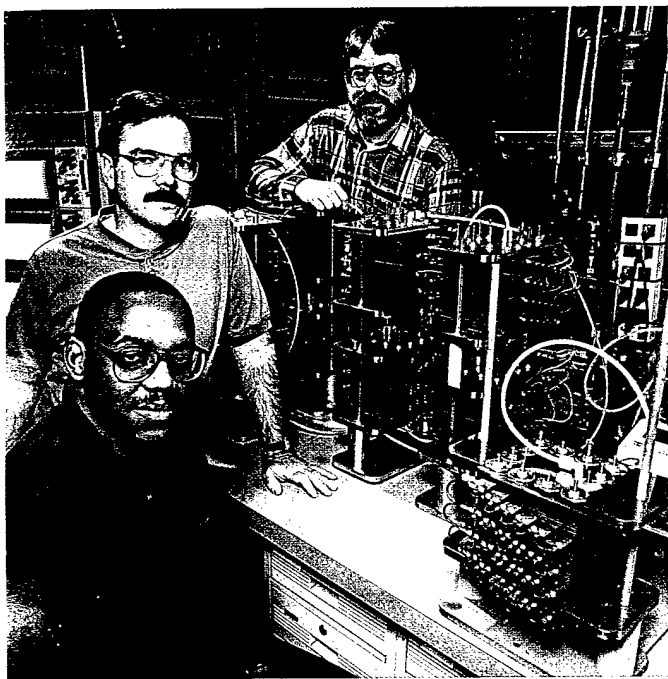
Aerogels exhibit many remarkable properties, such as the best electrical, thermal, and sound insulation of any known solid. As a result, few materials offer as many potential applications as aerogels. These versatile materials could be used in electronics, optics, thermal pane window inserts, solar panels, novelties, toys, jewelry, helmets, insulation tiles, refrigerators, freezers, and space applications and as catalysts for chemical reactions. Two recent advancements at the Laboratory in inorganic and organic aerogel process technology have improved the position of aerogels as a commercially viable technological innovation.



Getting Aerogels into Shape

A major stumbling block to the entry of silica aerogel products into the marketplace in a significant way is their high production cost, due mainly to a long and tedious manufacturing process. In response, a team of Laboratory researchers developed a process for producing shaped aerogel parts that is more than 30 times faster than conventional methods.

The injection molding process for net-shaped aerogels uses various molds (samples shown) to rapidly produce silica aerogels in a variety of shapes.



Pictured from left to right are Greg Mack, Joe Farmer, and Dave Fix, who developed the capacitive deionization process using carbon aerogel electrodes.

"We believe this process holds the key to making aerogels readily available for commercialization in many applications and prospective markets," says team leader and physicist Larry Hrubesh.

Hrubesh notes that silica aerogels are traditionally prepared by mixing a silicon-alkoxide— $\text{Si}(\text{OC}_x\text{H}_y)_4$ —compound with alcohol, water, and a small amount of ammonium hydroxide. When mixed, these ingredients form a gel containing a network of silicon dioxide particles. If the gel is carefully dried, an intact aerogel remains. The challenge is to heat the gel to remove the liquid without destroying the fragile silicon dioxide microstructure.

The problem facing manufacturers is that during drying, the liquid molecules exert a force called surface tension that pulls the gel in on itself and crushes the microstructure. One means of circumventing this problem is to carefully heat the liquid-filled gel under pressure until the liquid converts to a supercritical state. In this state, material behaves like neither a pure liquid nor a gas and no longer exerts surface tension. The fluid can then be directly removed from the gel without cracking it. However, this process, used by aerogel manufacturers, takes many hours to complete and is very costly.

The Livermore team's method eliminates the stress in the gel during conversion of the liquid to the supercritical state, dramatically speeding the drying process. The new process is very similar to injection molding, a common process used to manufacture certain types of plastics. First, the precursor chemicals are injected directly into a two-piece, sealed mold.

While the mold is rapidly heated, the liquids react to form the gel. The gel's shape is defined by the mold walls, which keep it from straining under the influence of the rising hydrostatic pressure of the liquid within the gel.

After as little as 20 minutes, the temperature and pressure within the mold produce the supercritical fluids, which are rapidly purged from the confined gel without cracking it. The internal pressure in the mold is rapidly lowered by releasing it through an opening. After the mold is cooled for a few minutes, it is opened and the finished aerogel part is removed. The entire process, from start to finish, takes minutes instead of the hours (or even days) required by conventional processes, and the finished aerogels require no further machining or polishing.

"Our process is the only one that can mass-produce aerogels of precise sizes and shapes while maintaining high surface tolerances," says Hrubesh. "This is possible because the mold totally defines the size, shape, and surface quality of the aerogel object. Other processes require at least one free surface, which can distort during supercritical drying."

The Livermore team estimates that the cost for aerogels made using the new process is less than \$3 per liter, as opposed to the \$25-per-liter price of aerogels available today using conventional processes. Furthermore, the new process reduces liquid waste by 40% over other methods because the liquid purged from the gel can be reused to make more aerogels. Finally, the process uses about 10 times less energy than other techniques because it does not require the pumping of fluids and because it heats the molds for only a short time.

The Livermore aerogel processing breakthrough was aided by the analytical work of Paul Coronado and John Poco, who fabricated double-wall containers with thermocouples inside to monitor the pressure and temperature of the gelling process. Using these containers led to a much greater understanding of aerogel chemistry and the discovery that changing small amounts of starting ingredients has a significant effect on the gelation and drying processes.

The research team is continuing to refine the injection molding process. Taking advantage of the new technology, team members are also making small double-paned windows containing a sealed layer of silica aerogel. Studies have shown that double-paned aerogel windows could have an insulating efficiency value of R-19, equivalent to the insulation value of a house wall backed with a 8.75-cm-thick roll of fiberglass.

Aerogels for Purifying

One of the most promising new applications for aerogels is in a cost-effective water purification process developed at the

Laboratory. The aerogel-based process can have a variety of uses ranging from extracting harmful contaminants from industrial waste water to desalinizing sea water. Known as carbon aerogel capacitive deionization (CDI), the patented process will probably consume less energy per unit of water purified than conventional technologies, does not require costly membranes or pumps, operates at ambient temperature, and is resistant to chemical attack.

The carbon aerogel CDI process works by sending solutions with various positively and negatively charged ions through an electrochemical cell consisting of numerous electrodes containing organic aerogels. Laboratory researchers Rick Pekala and John Poco fabricated the double-sided electrodes by gluing two sheets of a carbon aerogel composite to both sides of a titanium plate that serves as both an electrical current collector and a structural support.

The carbon aerogels have exceptionally high surface areas (400 to 1100 m²/g) for a total of about 3 million cm² per electrode. (A stack of 192 pairs of carbon aerogel electrodes, used in the most recent series of experiments, has a total surface area of about 1 billion cm², the equivalent of 25 acres.)

"The electrically conductive, monolithic sheets of carbon aerogel have exceptionally high specific surface area that can be exploited by CDI," notes Joe Farmer, project leader.

After application of a voltage between two adjacent carbon aerogel electrodes, cations (positively charged ions) and anions (negatively charged ions) are drawn toward the electrode's cathode and anode, respectively. These ions (in the case of sea water, mainly sodium and chlorine) are electrostatically removed from the water and held at the surfaces of the electrodes, leaving purified water. The trapped ions are then released into a separate stream of rinse water comprising 1% or less of the volume of the product water.

Farmer says carbon aerogel CDI may offer significant advantages over competing methods (such as reverse osmosis and ion exchange), which typically consume large amounts of energy, involve costly and often troublesome membranes or high-pressure pumps, and often generate large quantities of corrosive wastes that must then be specially treated. For example, ion exchange columns, commonly used to remove heavy metals and radioisotopes from waste water, require about 100 kg of acid to regenerate 1 kg of cation exchange resin. In contrast, carbon aerogel CDI regenerates its cells by electrically discharging them; the cells are then rinsed with a small volume of water.

One of the most interesting potential applications for carbon aerogel CDI is desalinization of brackish water (containing typically 800 to 3200 ppm salt) for residential, commercial, and agricultural purposes. Preliminary studies show that the process may require less energy than other

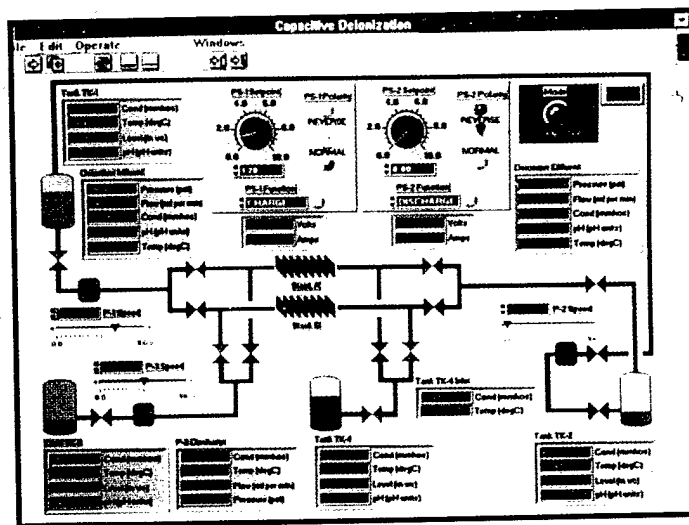
competing technologies because it does not employ complex membranes and does not require flow-through porous media. At some point in the future, it also may be possible to treat sea water (35,000 ppm salt), an important application for many parched California coast communities.

The Livermore research team experimented with various salt concentrations and operating voltages and showed that the system is capable of removing 95% of salt before the carbon aerogel electrodes become saturated. After several months of operation at 1.2 volts per cell (judged the most effective setting), the electrodes lost only 6 to 8% of their capacity.

Perhaps the most important application involves treating liquids containing radioisotopes. Farmer notes that both the U.S. Department of Energy and the former Soviet Union have large inventories of solutions contaminated with radioactive materials. Unlike ion exchange, carbon aerogel CDI can treat these radioactive wastes without generating secondary wastes.

Other potential applications for carbon aerogel CDI include the purification of boiler water for fossil and nuclear power plants, treatment of agricultural waste water containing pesticides and other toxic compounds, purification of water for semiconductor processing and other manufacturing processes, and treatment of waste water from electroplating operations.

Currently, LLNL is discussing plans with the U.S. Army to apply carbon aerogel CDI as part of an effort to destroy old stocks of mustard gas and for developing compact desalination units that can be stored for long periods without deteriorating. It is also holding talks with the U.S. Air Force about using carbon aerogel CDI to remove contaminants from rinse water generated from plating operations. Any commercialization of the technology will be done by private industry through appropriate agreements with the Laboratory.



Farmer says that more research and development is needed before carbon aerogel CDI technology can be incorporated into any large-scale plant. Future efforts will include component aging tests, more precise energy analyses, and experiments with high concentrations of various salts as well as acidic and basic electrolytes. Finally, cost-effective, high-volume production processes are needed for carbon aerogel electrodes to achieve their practical benefits.

For further information

Injection Molding Process for Net-Shaped Aerogels

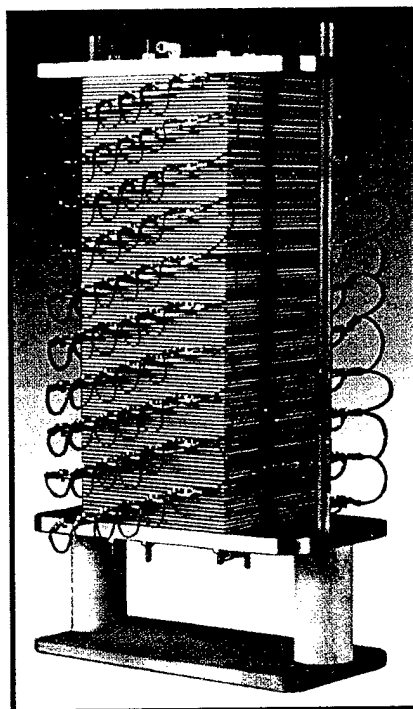
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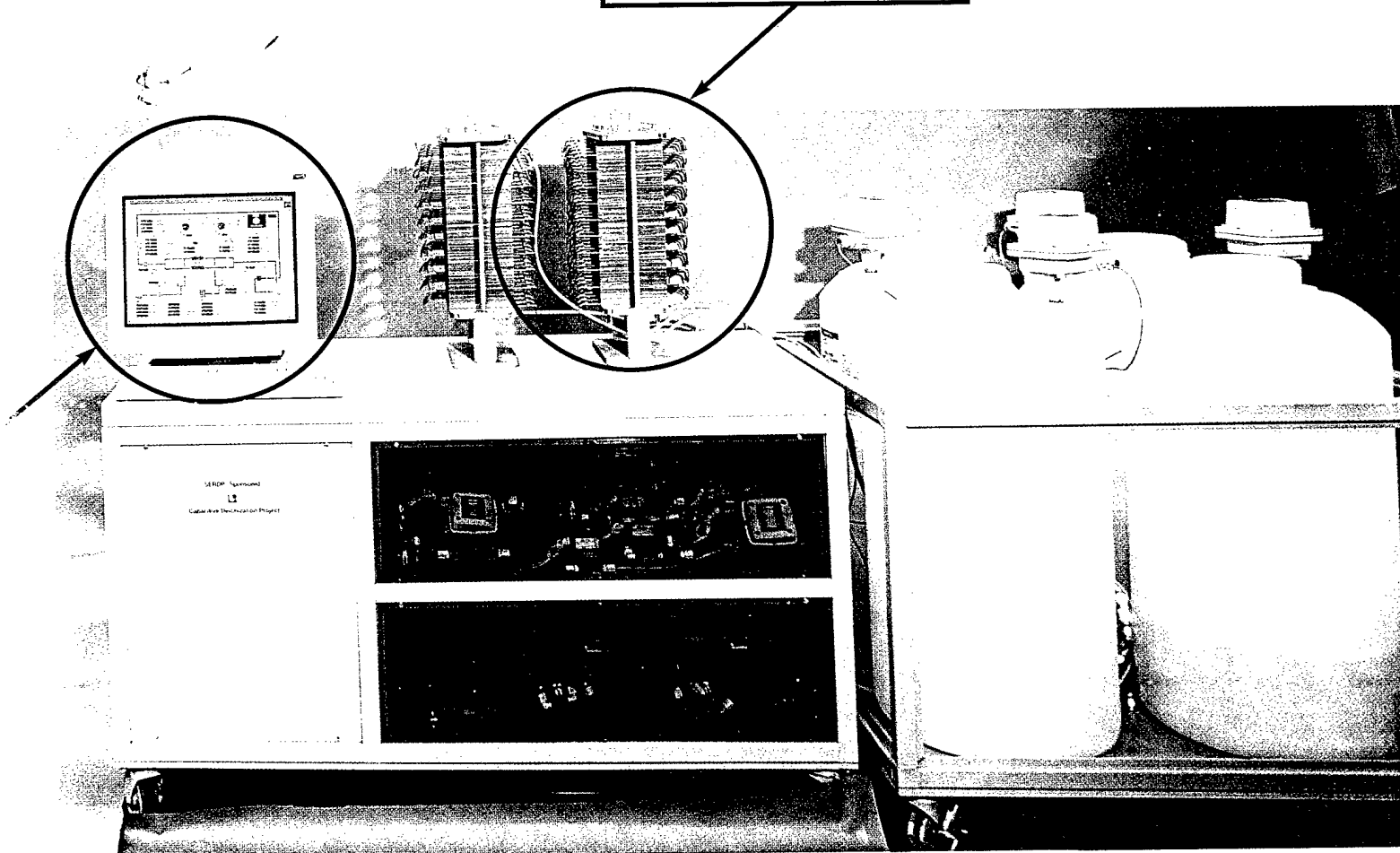
Carbon Aerogel Capacitive Deionization

Contact: Joseph C. Farmer

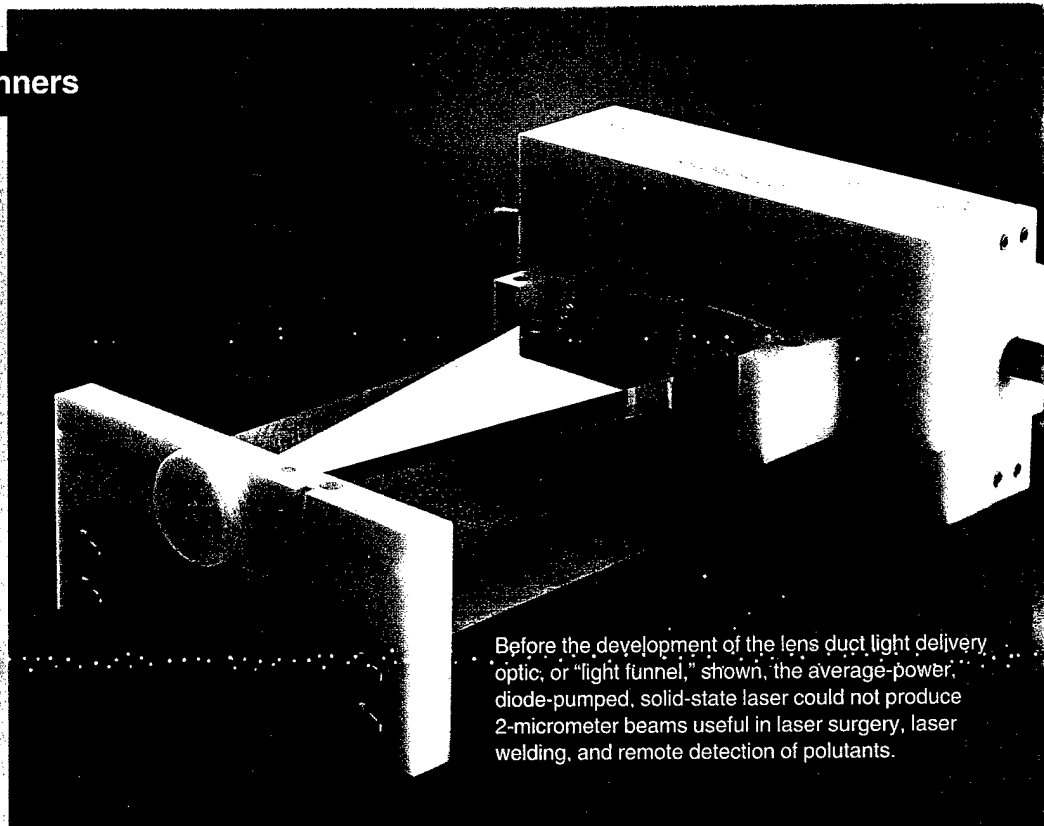
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All control of the fully automated, continuous-flow carbon aerogel capacitive deionization system shown is done with a "click of a mouse" on the control screen. At the left is a stack of carbon aerogel electrodes, the heart of the system.



A Light Funnel for Diode-Pumped, Solid-State Lasers



Before the development of the lens duct light delivery optic, or "light funnel," shown, the average-power, diode-pumped, solid-state laser could not produce 2-micrometer beams useful in laser surgery, laser welding, and remote detection of pollutants.

LASER beams do not just happen. Atoms must be pumped with energy to "excite" them. As excitation continues, the energy in the atoms increases, and the atoms give off energy in the form of intense light. The light can be visible light or invisible light like infrared or ultraviolet light. Depending on the type of laser, the excited atoms may be in a gas or a solid material, and the pumping mechanism may take a variety of forms.

Flashlamps have been used for excitation since the birth of lasers in the early 1960s. But flashlamps need to be replaced frequently, and they do not deliver energy very efficiently. So, about 15 years ago, scientists began to investigate the diode, a semiconductor device that converts electric power directly into light, as a pumping device for some solid-state lasers. The diode pump source began to be widely used about five years ago. Yet, diode-pumped lasers had drawbacks, too. While the diodes could produce laser light in high-intensity pulses, their overall power output was low (less than 1 kW/cm²), which reduced their usefulness.

A team of Laboratory scientists led by Ray Beach hit upon a remarkably simple solution to the low power output problem. "We imagined a 'light funnel,'" says Beach, "wherein the output from the diodes is captured and manipulated to concentrate the pump light." This light funnel is a small lens for concentrating, or "conditioning," the output radiation of the diodes.

With higher power outputs, other advances became possible. Previously, applications requiring high-average power were limited to a 1-micrometer (μm)-wavelength beam when using a diode-pumped, solid-state laser. But with this new lens technology, a broader range of solid materials can be

used that produce a variety of wavelengths, increasing the applications of these lasers. A 2-μm beam is even more useful. In surgery, for instance, a 1-μm beam causes collateral damage to tissue around surgical area. A 2-μm beam, on the other hand, cuts and cauterizes the surgical area while causing much less collateral damage to surrounding tissue. A 2-μm beam also has a variety of remote sensing applications—it can be mounted on an airplane to detect wind shear or placed in a suspected area to detect the byproducts of chemical or biological warfare.

With a light funnel lens, the output power of diode-pumped, solid-state lasers is increased by a factor of 250, and their unit cost is reduced by a factor of 100. "Our product offers a simple, inexpensive, and commercially attractive technique for scaling up the output power of laser systems," says Beach. "And this ability to scale up the laser's output power drastically reduces its price."

The Lens Conditions the Light

The three components of the Laboratory team's all-solid-state system are a laser diode pump array, the light funnel or lens duct, and a laser crystal. The lens duct (shown above) lies at the heart of this new design. It condenses and homogenizes the diverging light output from the diode array, coupling the diode light to the laser material at the lens duct's exit face.

The pie-shaped lens duct can be fabricated of inexpensive glass or plastic. Its longer input face and shorter output face are coated with an antireflective material for more efficient transmission of light. The other four sides are uncoated and prevent light from escaping by reflective wave guiding, which is produced by internal reflection of the light beam. While this

optical device is particularly applicable to coupling diode array pump light to solid-state laser materials, it can be applied to any situation that requires focusing a beam of light to produce a smaller beam of greater intensity. This lens duct can be used to scale up both pulsed and average-power laser systems. The team has developed a pulsed, 1- μm , diode-pumped, solid-state laser with an output pulse energy of 100,000 microjoules (μJ). The nearest competitor produces just 400 μJ of output energy. This 250-fold gain in output pulse energy also costs much less to produce: \$1 per microjoule compared to that competitor's \$100 per microjoule. Other systems on the market produce even less output pulse energy at a higher cost per microjoule. An average-power, 1- μm laser also performs markedly better than those of the competition. The Laboratory's system produces 13 W of output power, which is two and a half times what the nearest competitor can produce and at a much lower cost.

A 2- μm unit developed by the Laboratory team produces 25 W of average output power. A diode-pumped, solid-state laser producing this much power at the 2- μm wavelength was not possible before the development of the lens, and as a consequence, no comparable commercial system exists.

Applications from Medicine to Manufacturing

Applications for this new class of lasers abound in medicine, radar, manufacturing, and materials processing. The average-power, 2- μm system was developed as a surgical laser. The \$78-million-per-year medical laser market is presently dominated by flashlamp-pumped lasers, which could

be replaced by the far more reliable and efficient all-solid-state, diode-pumped versions.

The Laboratory team's pulsed, 1- μm unit is suitable for underwater illumination and sensing, and it is also being investigated for photon-assisted ignition in a new generation of jet turbine engines. In addition, it can be used for a lidar (laser radar) system in which laser beams are reflected and scattered off clouds, smog layers, and atmospheric discontinuities, and the return signals measured. All of these applications require a laser that is not only compact and reliable but produces significantly more pulse energy than is currently available.

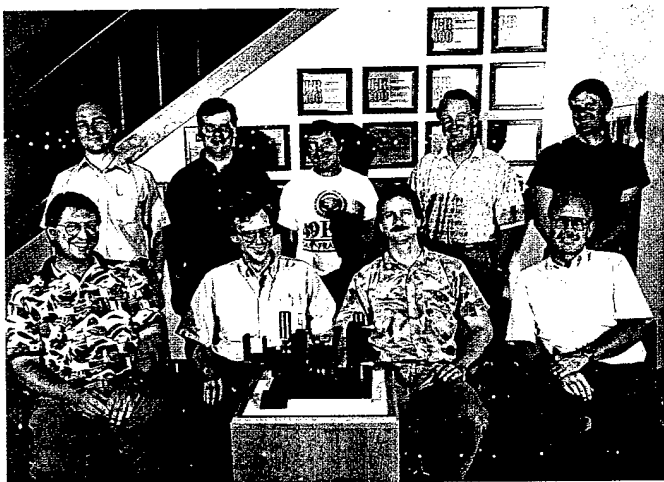
The Laboratory is developing a 2- μm system for use in treating infertility in humans and livestock. This laser drills tiny holes in the surfaces of human and animal eggs to increase their probability of being fertilized by sperm.

A high-average-power (100-W), solid-state laser is being evaluated as a replacement for the copper vapor lasers presently used by the U.S. Enrichment Corporation Laser Isotope Separation Project. Advantages of this laser over the copper vapor laser include higher efficiency, longer lifetime, and lower lifecycle costs.

The Livermore team is planning a kilowatt-class prototype for use in material processing applications such as metal cutting, welding, and hole cutting. For the past several years, the material processing community has embraced flashlamp-pumped lasers. But a drawback of those systems is that they are large and expensive to install and operate.

Looking even further into the future, megawatt-class lasers could be used for laser-driven, inertial confinement fusion power plants. In fact, Laboratory scientists developed one of their 1- μm systems as a subscale prototype for this application. Until very recently, scientists thought that solid-state lasers, even under ideal conditions, did not provide adequate efficiency and robustness for a commercial power plant setting. But by extending the current laser fusion technology base to include this lens duct technology and other advancements in solid-state lasers, a laser fusion driver concept appears feasible for the first time.

The diode-pumped, solid-state laser was developed under Cooperative Research and Development Agreements (CRADAs) with the Beckman Laser Institute and Medical Clinic at the University of California at Irvine, and Wellman Laboratories of Photomedicine, Boston, Massachusetts.



All-solid-state laser with diode-irradiance conditioning and the team that developed it. Back row from left to right: Steve Payne, Chris Marshall, John Lang, Chuck Petty, Scott Mitchell. Bottom row, left to right: Steve Mills, Ray Beach, Mark Emanuel, Bill Bennett.

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One of the World's Brightest Lasers

A powerful beam of green light shoots up into the night sky, beyond the Earth's atmosphere. The beam starts out pencil thin, and its divergence has been reduced almost to the limits that physical laws allow—it diverges to just a few meters in diameter after traveling hundreds of kilometers. The beam is precisely aimed to illuminate satellites, allowing researchers to identify and track them. Because of atmospheric distortions, the researchers cannot directly record clear images. But using the special capabilities of the laser generating this beam and a process known as high-resolution speckle imaging, they can collect a time-resolved "speckle" pattern and numerically reconstruct an undistorted image.

Another powerful beam of green laser light focuses on a jet of gas in a small vacuum chamber. With each pulse of light, a small ball of highly energized gas forms. This plasma converts the laser light into x rays that pass through an image-containing reticle and onto a silicon wafer coated with photoresist. The image on the reticle contains the ultra-fine circuit features of a very advanced computer chip. The x rays create shadows of the circuit features on the wafer and print them in the photoresist. The x rays generated by the laser represent a new commercial production technique for the next generation of integrated electronic circuits.

A single laser system developed by a team at the Laboratory enables both of these new applications. The laser produces high-energy pulses more frequently than any other laser system—30-joule (J) pulses six times a second (i.e., 6 hertz). This results in a peak power of over 2 gigawatts (GW) for each pulse and an average power of up to 180 W. This average power is 10 times greater than that of any commercially available solid-state laser in this pulse energy range. Even more importantly, the 2-GW peak power, which is equivalent to the peak output of a nuclear power plant, can be focused to a spot only a few micrometers in diameter, thanks to the low divergence of the beam. As Brent Dane, team leader for the Laboratory's effort on this laser, notes, "The combination of high average power and very low divergence has produced a world's record for average brightness for a solid-state laser."

While producing high-energy pulses, the laser can also operate at a broad range of pulse durations, from 10 to 1,000 nanoseconds (1 ns = 1 billionth of a second, or 1×10^{-9} s). Solid-state laser systems have typically operated in pulse durations of either 10 to 30 ns or more than 100,000 ns. In the



High-average-power solid-state laser developers (from left) Brent Dane, Lloyd Hackel, and Mary Norton with an advanced, two-amplifier prototype of their invention in the laboratory. Using recently improved amplifier glass developed with Schott Technologies, this laser should exceed the average power of the award-winning system by a factor of 4.

past, achieving pulse durations between 30 ns and 100,000 ns with a solid-state laser has been very difficult and has resulted in pulse energies of only a few millijoules.

A Unique Amplifier

A new type of laser amplifier consisting of mirrors, rotators, polarizers, special laser glass, and other optical devices is behind all of these "firsts." All materials used in the amplifier have high damage thresholds to withstand the extremely high energies.

The laser starts at an oscillator that produces a low-power, high-quality beam of invisible light with a narrow bandwidth. This beam then passes many times through the amplifier, its energy increased from millijoules to several tens of joules. A full transit from the oscillator to the amplifier's exit takes about 40 ns.

The amplifier incorporates a solid-state slab gain medium made of neodymium-doped glass (Nd:glass) with xenon flashlamps mounted around it. The flashlamps pulse rapidly, pumping light energy into the glass slab. The beam extracts this energy as it passes through the slab, generating an output pulse that has thousands of times more peak power than the flashlamp source.

The slab, designed and constructed at the Laboratory, has one thinner dimension to allow the heat to conduct out. The laser light "zig-zags" through the slab gain medium, internally reflecting off the side walls of the slab and averaging much of the thermal distortion within the slab. Hot spots in the slab, which cause optical distortion and could damage the gain medium, are virtually eliminated.

Most laser systems degrade during this amplification stage because of the high thermal loading placed on the gain medium. In this system, however, the team uses a device called a phase conjugator, which the beam enters midway through its transit in the amplifier. The stimulated Brillouin scattering (SBS) phase conjugator is a special mirror that reverses the beam's phase and corrects distortions. Although the flashlamp pump and the slab have been designed to reduce distortion, heat builds up during the multiple pulses of the flashlamps and distorts the laser beam wavefront. The phase conjugator reverses the sign of the beam distortions and returns the beam back through the amplifier for an equal number of passes, almost completely canceling the phase error and producing near-perfect beam quality and very stable beam pointing.

The phase conjugator is simply a glass cell with a quartz window. A lens focuses the input light into a liquid, carbon tetrachloride, that fills the cell. Through a mechanism called the electrostrictive effect, a density grating builds in the cell and reflects the beam with a reversed phase. No moving parts or electrical circuits are required. The technique reverses the wavefront with very high resolution without doing a single computer calculation or moving a single actuator.

Glass cells filled with carbon tetrachloride are not what we generally think of as mirrors. They are, in fact, a type of nonlinear mirror. SBS phase conjugators have been discussed

in scientific literature for many years and experimented with in laboratories. But this is the first instance of one put to practical use in a working, high-power laser system.

Another feature of this system is its ability to double the frequency of the output beam so that it changes from infrared light to visible, green light. Green light diverges less than infrared light, an important attribute for long-range uses where a beam with low divergence is needed.

To obtain even higher average power output from the laser, a new laser glass with much higher mechanical strength has been developed that could double the average power of a single amplifier. Furthermore, the use of SBS phase conjugation allows multiple, sequentially firing amplifier heads to be combined in a single laser beam train. A two-head laboratory prototype is currently in operation, and a version of the laser using four amplifier heads is under construction. The average power of the latter is expected to easily exceed 1,000 W.

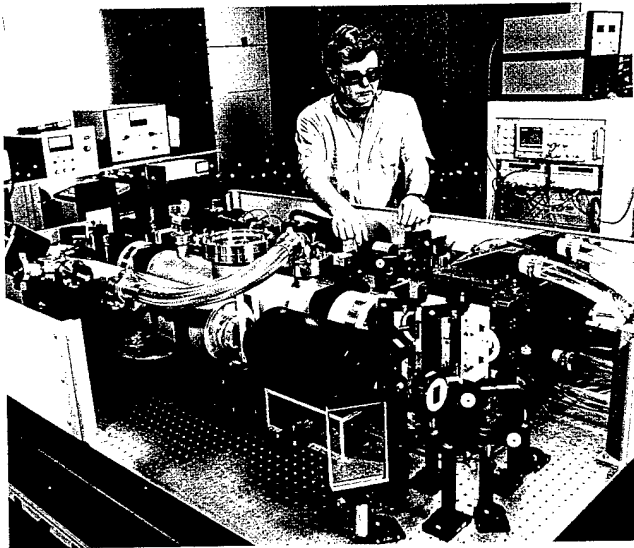
Lighting Launches and Removing Graffiti

High-resolution speckle imaging and x-ray lithography are just two applications for this new laser.

The frequency-doubled green light output of this system, with its narrow bandwidth and accurate long-range pointing stability, is ideal for advanced laser radar uses. For the U.S. Navy, the team developed a high-energy, green-output laser with a 500-ns pulse duration and delivered it to the Navy at Cape Canaveral, where it will be used to illuminate rocket launches from the Kennedy Space Center. During independent testing prior to shipment, the laser fired over one million shots with no maintenance or realignment required.

A version of the laser, operating at around 100 joules per pulse, could provide the pump source for a tabletop-size x-ray laser, whose coherent output would be used to produce very-high-resolution, three-dimensional images.

The team has also discovered that high-energy, short-pulse lasers are excellent for quickly removing paint from surfaces with an intense acoustic wave that does not harm the surface beneath the paint. This is an environmentally sound method of removing paint from aircraft and ships, lead-based paint from public and private buildings, and painted graffiti from a variety of surfaces. "Graffiti removal is a hot topic," notes Dane, "and is being explored further by other scientists at the Laboratory."



Electrooptics technician James Wintemute completes final alignment of a packaged laser system. Several of these systems have been installed and are in use, one of them at Kennedy Space Center in Florida, where it will be used to illuminate rocket launches.

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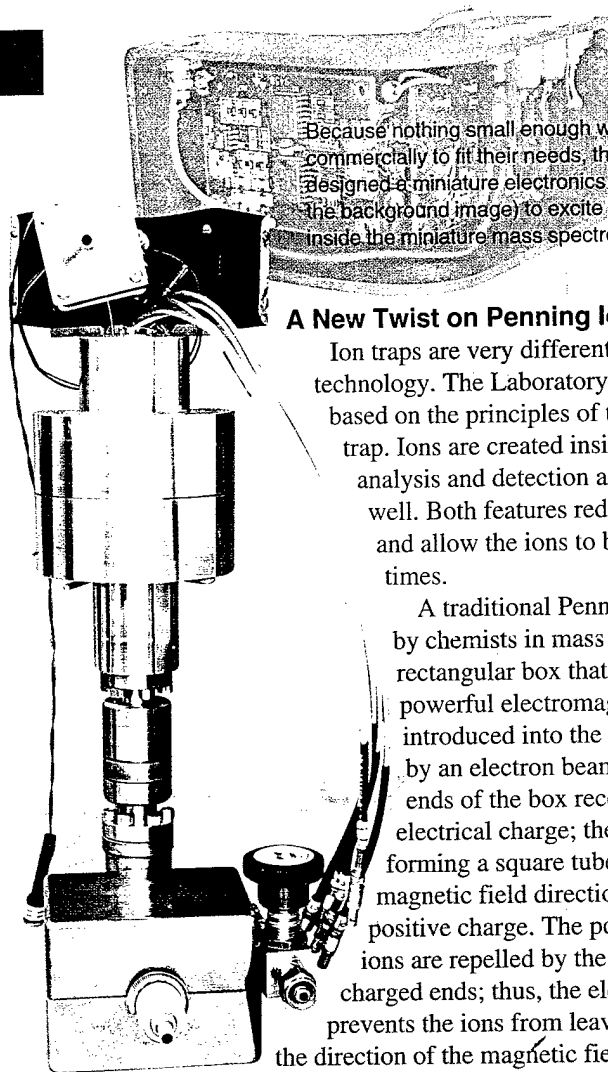
A Miniature Mass Spectrometer

CHEMISTS and forensic scientists have been using mass spectrometers for decades to analyze a variety of sample materials. In the early 1980s, atomic physicists began to use a highly sensitive type of mass spectrometer called an ion trap to study individual particles. Some research ion traps are sufficiently sensitive to measure one ion at a time with a mass accuracy of one in one quintillion, or 1 in 1,000,000,000,000,000 (1:10¹⁸).

Dan Dietrich, a senior researcher at the Laboratory, and his colleague, Bob Keville, had been using these ion traps for several years, and began to wonder whether the theory behind them could be applied to a smaller commercial mass spectrometer. Small fixed or portable mass spectrometers would be immensely useful in a variety of situations—as a remote detection device for monitoring air quality or for sniffing out drugs or compounds related to nuclear or chemical weapons.

The central detection device in research mass spectrometers is very small, which is what Dietrich and Keville found attractive. But the accompanying equipment fills a room. So, they developed the first truly hand-portable mass spectrometer, which fits into a small briefcase. It weighs just 12.3 kg (33 lb) and operates off a battery. Its accuracy is 1 in 1,000 (1:10³) and will ultimately be 1:10⁴, which is a long way from the accuracy of the large research units but sufficient for the uses planned for it.

Although this miniature mass spectrometer is still under development, it could have greater sensitivity and efficiency than conventional laboratory-based, single-pass mass spectrometers. In a single-pass unit, there are an ion source where the sampling material is ionized, an analyzer where the ions are separated in space according to their charge, mass, and velocity, and a detector that measures the number of ions analyzed. Some loss of ions as they move from region to region within this type of unit is inevitable.



Because nothing small enough was available commercially to fit their needs, the Livermore team designed a miniature electronics package (shown in the background image) to excite and detect ions inside the miniature mass spectrometer's ion trap.

A New Twist on Penning Ion Traps

Ion traps are very different from single-pass technology. The Laboratory's new unit is based on the principles of the Penning ion trap. Ions are created inside the trap, and analysis and detection are done there as well. Both features reduce ion losses and allow the ions to be sampled many times.

A traditional Penning ion trap used by chemists in mass spectrometry is a rectangular box that sits inside a powerful electromagnet. A gas is introduced into the box and ionized by an electron beam. Two opposing ends of the box receive a positive electrical charge; the four sides, forming a square tube along the magnetic field direction, receive a less positive charge. The positively charged ions are repelled by the positively charged ends; thus, the electrical voltage prevents the ions from leaving the box along the direction of the magnetic field. If the positive ions try to leave the box across the direction of the magnetic field, i.e., toward any of the less positive sides, they are deflected by the magnetic field into cyclical orbits. This motion is called cyclotron motion. The frequency of this cyclical motion is determined only by the strength of the magnetic field and the ratio of the ions' electrical charge to their mass. By rapidly reversing the sign of the voltage on opposing pairs of these less positive sides, the turning ions can be pulled into orbits optimized for detection. Because the values of the magnetic field and the ions' electrical charge are known, the mass of a "trapped" ion can be determined from the frequency of its orbit. This method of determining an ion's molecular weight and thus its identity is known as ion cyclotron resonance and is the operating principle of the Penning ion trap.

High resolution ion traps developed for use by atomic physicists use the basic principles of the rectangular trap but are open-ended cylinders that fit inside powerful superconducting magnets. In a cylindrical ion trap, the homogeneous magnetic field keeps the ions in a very narrow orbit in the middle of the cylinder. The combination of huge magnets and ultra-high vacuums, which can keep individual

ions in orbit inside the trap for weeks, is what allows these ion traps to be so extraordinarily accurate.

While the ion trap devised by the Laboratory team is about the same size as the trap in the research units, the magnetic field is provided by a small permanent magnet, greatly reducing the overall size and power requirements of the unit. The design of the trap, the electron-beam, the vacuum system, and the inlet valve are also new. The goal is an ion residence time of 100 milliseconds, which provides sufficient resolution to detect trace samples of contaminants or other compounds in air.

The permanent magnet is designed to optimize magnetic field. It is cylindrical, slightly less than 7.6 cm across and 5.7 cm high, with a center bore of slightly less than 2.5 cm. A thin-walled vacuum chamber containing the ion trap fits inside the hole. A small ion pump, along with a miniature cryogenic pump, maintains the tube's vacuum pressure. A miniature Piezo electric inlet valve admits gas to be sampled into the tube. There is also a pulsed electron source beam that ionizes the gas in the trap.

Ions are confined in a volume defined by electrodes that are plated on the inner diameter of the vacuum tube. Changing voltages on the electrodes excites the ions into their cyclotron orbit within the trap. Between the excitation electrodes are the detection electrodes that sense the ions' orbital frequency and thus the mass and identity of the ions in the trap.



Dan Dietrich (left) and Bob Keville with their miniature mass spectrometer.

Several innovations in our ion trap keep power consumption very low—to about half a watt. (That figure does not include the 20 W needed by the laptop computer that accompanies the device.) First, the permanent magnet is the most obvious power saver. Second, smaller is better. The smaller the trap is—that is, the closer the particles are to the detection electrodes—the greater the trap's sensitivity and resolution are and the lower the electrical power consumption. Next, the unique design of the inlet valve is important in reducing energy consumption. In most mass spectrometers, gas is continually bled in, and thus, large pumps are needed to maintain the vacuum in the spectrometer. This new miniature Piezo electric inlet valve lets in gas in small pulses, reducing vacuum pumping requirements. Finally, the vacuum pump itself is also a new power-saving design.

Uses in the Field

Research mass spectrometers used in laboratories are complex, bench-top instruments requiring a highly trained operator. Our inexpensive and portable mass spectrometer, in contrast, will find numerous applications outside of the laboratory.

These units can act as air quality monitors in a closed or confined space, such as a factory where chemical weapons manufacture is suspected. Someday, with additional front-end filters or sensors, they could be used to indicate the presence of air-borne disease agents. In a home, office, or factory, they could be incorporated into feedback control loops and alarm systems to warn of hazardous conditions. A home sensor could monitor freon and radon as well as carbon monoxide, carbon dioxide, methane, propane, and other hydrocarbons.

Law enforcement agencies could replace breathalyzers and drug- and explosives-sniffing dogs with these sensors. Testing drivers for alcohol use or sniffing out drugs could be performed remotely, which would mean increased safety for police officers and other officials.

Industries with critical process control functions could monitor for sensitive manufacturing byproducts such as chlorofluorocarbons, hydrazine, helium, nitrous oxide, nitrous dioxide, and sulfuric acid. Even small industrial accidents could be avoided with these sensors in place.

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More Efficient, Less Expensive Electron Beam Processing

ENVIRONMENTAL concerns affect almost every industry. Thermal methods of curing inks, adhesives, and coatings produce volatile organic compounds (VOCs), which are considered hazardous materials. So polymers that can be cured using ultraviolet light and electron beams, which do not produce VOCs, have been developed. Although electron beam processing has many advantages over ultraviolet processing, the high cost and complexity of commercial electron beam equipment has limited its use to less than 1% of the multibillion-dollar radiation-curable materials market, which is now growing at more than 20% per year.

Thousands of ultraviolet-curable products and applications are developed every year, but concerns are being raised because their formulations contain highly toxic photoinitiators, which can cause numerous health problems. On the other hand, electron-beam-curable materials are much less toxic, cure independent of color, and use 3 to 7 times less energy than ultraviolet curing equipment. But conventional electron beam equipment could never compete with ultraviolet due to its large size and astronomical cost.

That could change if industry puts to use a new product developed by scientists at the Laboratory and American International Technologies Inc. (AIT) of Torrance, California. Together they developed a low-cost electron beam gun in a vacuum tube as a replacement for these expensive systems. A system using sealed tubes costs ten times less than conventional electron beam systems of similar power, is smaller and easier to use, and increases worker safety by reducing exposure to x rays and high electrical voltages.

Booth Myers, technical leader of the Laboratory portion of the team, says, "Despite the high cost of existing systems, electron beam processing is a growing field, driven by industry needs to avoid the use of VOCs in ink, adhesive, and paint curing. Our lower cost electron beam gun is not only competitive for existing applications, but will open up numerous new uses for electron beam processing. In addition,



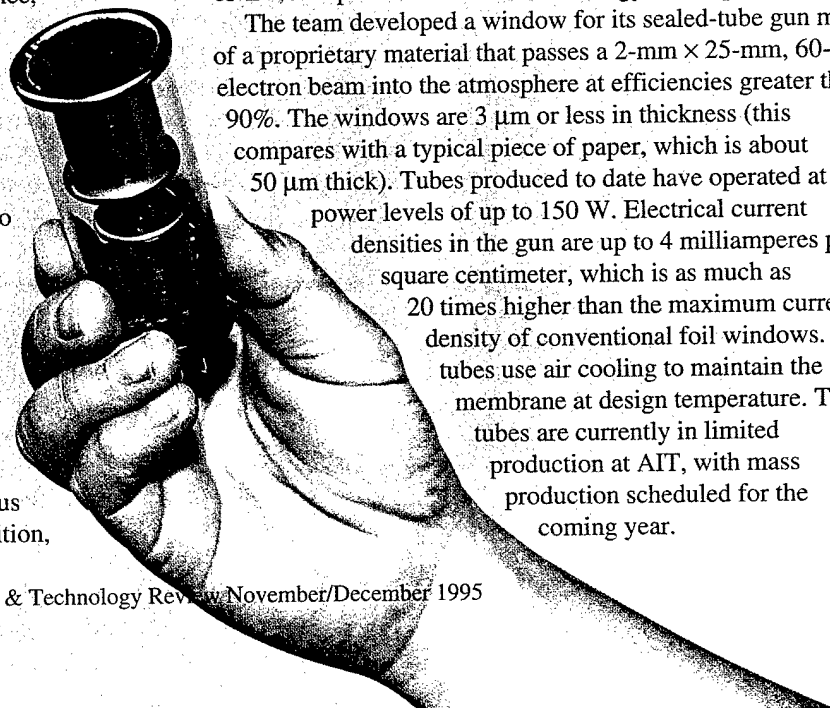
The sealed-tub electron beam gun can generate 150 W of electron beam power.

the ability to mass produce these tubes makes it possible to compete with ultraviolet systems in both size and cost."

The New Window

The large vacuum vessels used in conventional electron beam systems have titanium or aluminum foil windows through which the beam exits into the atmosphere, where the processing takes place. Typical current densities for metal foils are 200 microamperes per square centimeter at 150 to 300 kilovolts (kV). The minimum voltage of 150 kV is significant because that is the energy required just to push the electrons through the metal foil window. So for coatings less than 40 micrometers (μm) thick (a typical thickness for ink, paint, or adhesive coatings), these conventional systems are very inefficient because more energy is deposited into the metal foil window than into the coating being cured. For example, a 300-kV accelerator deposits less than 5% of its beam energy on a 40- μm -thick polymer. Because most of the emerging electron beam processing applications are for thin films, this inefficiency is a significant problem. The sealed-tube electron beam gun developed by the LLNL/AIT team offers vastly increased electrical efficiency—operating at 65 kV, it deposits over 75% of its energy on the polymer.

The team developed a window for its sealed-tube gun made of a proprietary material that passes a 2-mm \times 25-mm, 60-kV electron beam into the atmosphere at efficiencies greater than 90%. The windows are 3 μm or less in thickness (this compares with a typical piece of paper, which is about 50 μm thick). Tubes produced to date have operated at power levels of up to 150 W. Electrical current densities in the gun are up to 4 milliamperes per square centimeter, which is as much as 20 times higher than the maximum current density of conventional foil windows. The tubes use air cooling to maintain the membrane at design temperature. The tubes are currently in limited production at AIT, with mass production scheduled for the coming year.





Pictured from left to right are Hao-Lin Chen, Booth Myers, Glenn Meyer, James Davin, and Dino Ciarlo. At the bottom are American International Technologies Inc. team members George Wakalopoulos, President of AIT, and Peter Bond.

Smaller, Safer, Less Expensive

Depending on the power and features selected, the initial cost of a conventional system ranges from \$300,000 to \$2 million. A comparable sealed-tube unit and supporting equipment will cost \$25,000 to \$100,000, depending on the power. Annual operating costs for existing systems are also high, while the new units will operate at one-tenth that cost. The new systems are less than one-tenth the size of a comparable conventional system and require very little maintenance, which should yield additional cost savings. Existing systems are large, fixed, custom-designed installations, uneconomical for small-scale, lower power applications. The new tubes can be used alone or in groups, and a wide variety of processes and applications can be addressed in a variety of configurations.

Operation at lower voltages, which is possible because of the new thin-film window, greatly reduces x-ray generation and improves worker safety. It is also more efficient for processing thin material layers such as inks, adhesives, and paints and for surface sterilization, because the reduced penetration depth of lower energy electrons is better matched to the thickness of the material being processed.

Conventional systems require periodic replacement of the foil windows after about 2,000 hours of use. This process demands extensive disassembly of the unit. The new sealed tubes can be unplugged after 2,000 hours of use and replaced with new ones, reducing unit downtime from days to minutes. In fact, laboratory tests and calculations show that lifetimes of 7,000 hours are feasible, which would reduce operating costs even further.

Expanding the Range of Uses

Currently, the primary commercial application for electron beam processing is curing polymers without using VOCs.

Because electron beam polymerization does not produce VOCs and does not use the toxic photoinitiators used in ultraviolet light curing, it satisfies many of the existing and anticipated environmental restrictions on manufacturing processes.

Electron beam processing is also used to sterilize medical supplies and food packaging materials that cannot tolerate sterilization by high temperature or chemicals. Electron beam sterilization of food has not gained widespread acceptance in the U.S., although its use is increasing in other countries.

The availability of a low-cost, self-contained source of high-energy electrons expands enormously the range of uses for electron beam radiation. Some examples include:

- Fast, on-line chemical and elemental analysis. Electron beams induce visible, ultraviolet, and x-ray fluorescence whose spectrum identifies the elements in the irradiated sample. "Since this process is effectively instantaneous, it will likely find many applications both for on-line industrial monitoring as well as laboratory analysis," notes Myers.
- Injection of electrons into liquid sprays. The addition of electrons greatly improves spray effectiveness by reducing the average droplet size formed at the nozzle. Applications include pesticide spraying, turbine fuel injection, and internal combustion engine fuel injection.
- Destruction of toxic industrial compounds or cleanup of stack or automotive exhaust gases. Because electron beams are known to decompose both organic and inorganic compounds, they could be used as an economical method of waste treatment without incineration. The government might find this application useful for site cleanup and restoration programs. Another application of this new technology, to disassemble retired nuclear weapons, is under evaluation.
- Generation of ozone. Ozone is gaining popularity as a purifier for water supply systems, either instead of or in addition to chlorine. Ozone generation with electron beams is known to be approximately twice as energy efficient as conventional means using electrical discharge devices.

The electron beam gun described in this article was developed under a Cooperative Research and Development Agreement (CRADA) with American International Technologies Inc., Torrance, California. For further information about commercial applications, contact AIT at (310) 328-3484.

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